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## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<b>(51) International Patent Classification <sup>6</sup> :</b>  <b>A61K 7/06, 7/50</b>	<b>A1</b>	<b>(11) International Publication Number:</b> <b>WO 99/49836</b>  <b>(43) International Publication Date:</b> 7 October 1999 (07.10.99)
<b>(21) International Application Number:</b> PCT/EP99/01902  <b>(22) International Filing Date:</b> 22 March 1999 (22.03.99)  <b>(30) Priority Data:</b> 9806826.5                      30 March 1998 (30.03.98)                      GB  <b>(71) Applicant (for AE AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZA ZW only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB).  <b>(71) Applicant (for all designated States except AE AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZA ZW):</b> UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).  <b>(71) Applicant (for IN only):</b> HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020, Maharashtra (IN).  <b>(72) Inventor:</b> MURRAY, Andrew, Malcolm; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB).		<b>(74) Agent:</b> BUTLER, David, John; Unilever plc, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB).  <b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> HAIR CONDITIONING COMPOSITIONS  <b>(57) Abstract</b>  An aqueous hair conditioning composition comprising, in addition to water: i) at least one conditioning surfactant, and ii) emulsified particles of an amino functional silicone of the general formula: $\text{Si}(\text{CH}_3)_3\text{--O--}[\text{Si}(\text{CH}_3)_2\text{--O--}]_x\text{--}[\text{Si}(\text{CH}_3)(\text{R--NH--CH}_2\text{CH}_2\text{NH}_2)\text{--O--}]_y\text{--Si}(\text{CH}_3)_3$ wherein $x + y$ is a number from about 50 to about 500, and wherein R is an alkylene group having from 2 to 5 carbon atoms; in which the amino functional silicone has a mole percent amino functionality of at least 1 mole %.		

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## HAIR CONDITIONING COMPOSITIONS

### 5 FIELD OF THE INVENTION

This invention relates to hair conditioning compositions intended to be rinsed off. In particular, the invention relates to hair conditioning compositions containing  
10 emulsified particles of amino functional silicone, which compositions condition the hair leaving it softer and more manageable.

### BACKGROUND AND PRIOR ART

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The use of silicones as conditioning agents in cosmetic formulations is well known and widely documented in the patent literature. Hair treatment compositions containing amino functional polysiloxanes have also been described in  
20 several publications. For example, U.S. 4,563,347 teaches that an aqueous emulsion of aminoalkyl substituted polydimethylsiloxane is useful to condition hair because it facilitates combing and imparts a smooth feel to hair. Other hair treating compositions containing amino functional  
25 polysiloxanes are described in U.S. 4,586,518, U.S. 4,601,902 and U.S. 4,618,819. Amino functional microemulsions have also been described in the field of hair care. For example, U.S. 4,749,732 describes hair care uses of polydiorganosiloxanes containing aminoalkyl groups

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modified by alkoxycarbonylalkyl substituents. U.S. 4,620,878 describes generally the preparation of emulsions of silicones containing polar substituents, and teaches a method of preparing clear microemulsions of amine functional polyorganosiloxanes, which can be mixed with a shampoo base of sodium lauryl ether sulphate and water to produce a stable, clear composition.

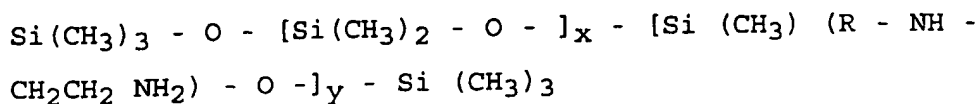
A problem encountered with hair conditioning formulations incorporating amino functional silicones is that the delivery of conditioning performance may be insufficient for many people, particularly in regions such as Japan and South East Asia where consumers desire a high level of conditioning and a "weighty" feel to their hair.

We have now found that the inclusion in a hair conditioning composition of a particular class of amino functional silicone, with specified mole percent amino functionality, significantly improves wet and dry conditioning performance.

#### SUMMARY OF THE INVENTION

The invention provides an aqueous hair conditioning composition comprising, in addition to water:

- i) at least one conditioning surfactant, and
- ii) emulsified particles of an amino functional silicone of general formula:



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wherein  $x + y$  is a number from about 50 to about 500, and wherein R is an alkylene group having from 2 to 5 carbon atoms;

- 5 in which the amino functional silicone has a mole percent amino functionality of at least 1 mole %.

#### DETAILED DESCRIPTION OF THE INVENTION

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##### Conditioning Surfactant

The composition according to the invention comprises one or more conditioning surfactants which are cosmetically  
15 acceptable and suitable for topical application to the hair.

Suitable conditioning surfactants are selected from cationic surfactants, used singly or in admixture. Examples include quaternary ammonium hydroxides or salts thereof, e.g  
20 chlorides.

Suitable cationic surfactants for use in hair conditioning compositions of the invention include cetyltrimethylammonium chloride, behenyltrimethylammonium chloride, cetylpyridinium  
25 chloride, tetramethylammonium chloride, tetraethylammonium chloride, octyltrimethylammonium chloride, dodecyltrimethylammonium chloride, hexadecyltrimethylammonium chloride, octyldimethylbenzylammonium chloride, decyldimethylbenzylammonium chloride,  
30 stearyldimethylbenzylammonium chloride, didodecyldimethylammonium chloride, dioctadecyldimethylammonium chloride, tallowtrimethylammonium chloride, cocotrimethylammonium chloride, and the

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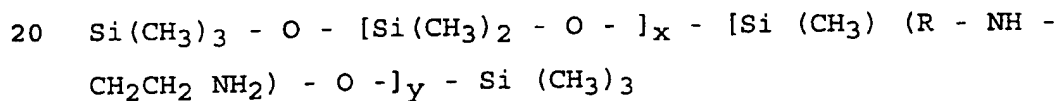
corresponding hydroxides thereof. Further suitable cationic surfactants include those materials having the CTFA designations Quaternium-5, Quaternium-31 and Quaternium-18. Mixtures of any of the foregoing materials may also be  
5 suitable. Particularly useful cationic surfactant for use in hair conditioners of the invention are cetyltrimethylammonium chloride and behenyltrimethylammonium chloride

In hair conditioning compositions of the invention, the level  
10 of cationic surfactant is preferably from 0.01 to 10%, more preferably 0.05 to 5%, most preferably 0.1 to 2% by weight based on total weight of the composition.

Emulsified amino functionalised silicone

15

Hair conditioning compositions according to the invention include emulsified particles of an amino functionalised silicone of general formula:



wherein  $x + y$  is a number from about 50 to about 500, and  
wherein R is an alkylene group having from 2 to 5 carbon  
25 atoms.

Preferably, the number  $x + y$  is in the range of from about 100 to about 300.

30 The amino functional silicone is insoluble in the aqueous matrix of the hair conditioning composition and so is present in an emulsified form, with the silicone present as dispersed particles.

- 5 -

We have found that amino functional silicones suitable for use in the invention need to have a mole percent amino functionality of at least 1.0 mole %. Suitably the mole percent amino functionality of the amino functional silicone ranges from about 1 to about 8.0 mole %, preferably from about 1 to about 5.0 mole %, such as about 1.7 mole %.

Various methods of making emulsions of particles of amino functional silicones for use in the invention are available and are well known and documented in the art. For example, emulsions may be prepared by high shear mechanical mixing of the silicone and water, or by emulsifying the silicone with water and an emulsifier (mixing the silicone into a heated solution of the emulsifier for instance), or by a combination of mechanical and chemical emulsification. A further suitable technique for preparation of emulsions of particles of silicones is emulsion polymerisation. Emulsion polymerised silicones as such are described in US 2 891 820 (Hyde), US 3 294 725 (Findlay) and US 3 360 491 (Axon).

The viscosity of the amino functional silicone itself (not the emulsion or the final hair conditioning composition) is not particularly critical and can suitably range from about 50 to 500,000 cst. Viscosity can be measured by means of a glass capillary viscometer as set out further in Dow Corning Corporate Test Method CTM004 July 20 1970.

Preferably the average silicone particle size of the emulsified particles of amino functional silicone in the hair conditioning composition is less than 20, preferably less than 10 microns. We have found that reducing the particle size generally improves conditioning performance. Most preferably the average amino functional

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silicone particle size in the hair conditioning composition is less than 2 microns, ideally it ranges from 0.01 to 1 micron. Silicone emulsions having an average silicone particle size of  $\leq 0.15$  microns are generally termed  
5 microemulsions.

Particle size may be measured by means of a laser light scattering technique, using a 2600D Particle Sizer from Malvern Instruments.

10

Specific examples of amino functional silicones suitable for use in the invention are the aminosilicone oils DC2-8220, DC2-8166, DC2-8466, and DC2-8950-114 (all ex Dow Corning),  
15 and GE 1149-75, (ex General Electric Silicones).

Also suitable are pre-formed emulsions of amino functional silicone oils with non ionic and/or cationic surfactant. This is particularly preferred since the pre-formed emulsion  
20 can be incorporated into the hair conditioning composition by simple mixing.

Pre-formed emulsions of amino functionalised silicone are available from suppliers of silicone oils such as Dow  
25 Corning, General Electric, Union Carbide, Wacker Chemie, Shin Etsu, Toshiba, Toyo Beauty Co, and Toray Silicone Co. Examples include emulsions DC2-8320, DC2-8306, DC2-8177 and DC2-8467, all available from Dow Corning.

30 Optional Ingredients

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- Emulsified, non-amino functionalised silicone

Hair conditioning compositions of the invention may suitably also comprise emulsified particles of a non-amino  
5 functionalised silicone, which is insoluble in the aqueous matrix of the composition and so is present in an emulsified form, with the silicone present as dispersed particles.

Suitable non-amino functionalised silicones include  
10 polydiorganosiloxanes, in particular polydimethylsiloxanes which have the CTFA designation dimethicone. Also suitable for use in hair conditioning compositions of the invention are polydimethyl siloxanes having hydroxyl end groups, which have the CTFA designation dimethiconol. Also suitable for  
15 use in hair conditioning compositions of the invention are silicone gums having a slight degree of cross-linking, as are described for example in WO 96/31188. These materials can impart body, volume and stylability to hair, as well as good wet and dry conditioning.

20 The viscosity of the emulsified non-amino functionalised silicone itself (not the emulsion or the final hair conditioning composition) is typically at least 10,000 cst. In general we have found that conditioning performance  
25 increases with increased viscosity. Accordingly, the viscosity of the silicone itself is preferably at least 60,000 cst, most preferably at least 500,000 cst, ideally at least 1,000,000 cst. Preferably the viscosity does not exceed  $10^9$  cst for ease of formulation.

30 Emulsified non-amino functional silicones for use in hair conditioning compositions of the invention will typically have an average silicone particle size in the composition of

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less than 30, preferably less than 20, more preferably less than 10 microns. Again, we have found that reducing the particle size generally improves conditioning performance. Most preferably the average silicone particle size of the emulsified non-amino functional silicone in the composition is less than 2 microns, ideally it ranges from 0.01 to 1 micron.

Suitable non-amino functional silicone emulsions for use in the invention are also commercially available in a pre-emulsified form.

Examples of suitable pre-formed emulsions include emulsions DC2-1766, DC2-1784, and microemulsions DC2-1865 and DC2-1870, all available from Dow Corning. These are all emulsions/microemulsions of dimethiconol. Cross-linked silicone gums are also available in a pre-emulsified form, which is advantageous for ease of formulation. A preferred example is the material available from Dow Corning as DC X2-1787, which is an emulsion of cross-linked dimethiconol gum. A further preferred example is the material available from Dow Corning as DC X2-1391, which is a microemulsion of cross-linked dimethiconol gum.

The total amount of silicone (amino functional and non-amino functional, if present) incorporated into the hair conditioning compositions of the invention depends on the level of conditioning desired and the material used. A preferred amount is from 0.01 to about 10% by weight of the total composition although these limits are not absolute. The lower limit is determined by the minimum level to achieve conditioning and the upper limit by the maximum

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level to avoid making the hair and/or skin unacceptably greasy.

We have found that a total amount of silicone of from 0.3 to 5 5%, preferably 0.5 to 3%, by weight of the total composition is a suitable level.

- Fatty Alcohol

10 Hair conditioning compositions of the invention advantageously incorporate a fatty alcohol material. The combined use of fatty alcohol materials and cationic surfactants in conditioning compositions is believed to be especially advantageous, because this leads to the formation  
15 of a lamellar phase, in which the cationic surfactant is dispersed.

Representative fatty alcohols comprise from 8 to 22 carbon atoms, more preferably 16 to 20. Examples of suitable fatty  
20 alcohols include cetyl alcohol, stearyl alcohol and mixtures thereof. The use of these materials is also advantageous in that they contribute to the overall conditioning properties of compositions of the invention.

25 Alkoxylated, (e.g. ethoxylated or propoxylated) fatty alcohols having from about 12 to about 18 carbon atoms in the alkyl chain can be used in place of, or in addition to, the fatty alcohols themselves. Suitable examples include ethylene glycol cetyl ether, polyoxyethylene (2) stearyl  
30 ether, polyoxyethylene (24) cetyl ether, and mixtures thereof.

- 10 -

The level of fatty alcohol and/or alkoxylated fatty alcohol material in conditioners of the invention is conveniently from 0.01 to 10%, preferably from 0.1 to 5% by weight of the composition.

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The weight ratio of cationic surfactant to fatty alcohol and/or alkoxylated fatty alcohol is suitably from 10:1 to 1:10, preferably from 4:1 to 1:8, optimally from 1:1 to 1:4.

10    -    Other optional ingredients

Compositions of this invention may contain any other ingredient normally used in hair treatment formulations. These other ingredients may include viscosity modifiers, preservatives, colouring agents, polyols such as glycerine and polypropylene glycol, chelating agents such as EDTA, antioxidants, fragrances, and sunscreens. Each of these ingredients will be present in an amount effective to accomplish its purpose. Generally these optional ingredients are included individually at a level of up to about 5% by weight of the total composition.

Preferably, compositions of this invention also contain adjuvants suitable for hair care. Generally such ingredients are included individually at a level of up to 2%, preferably up to 1%, by weight of the total composition.

Among suitable hair care adjuvants, are:

- 30    (i) natural hair root nutrients, such as amino acids and sugars. Examples of suitable amino acids include arginine, cysteine, glutamine, glutamic acid, isoleucine, leucine, methionine, serine and valine, and/or precursors and

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derivatives thereof. The amino acids may be added singly, in mixtures, or in the form of peptides, e.g. di- and tripeptides. The amino acids may also be added in the form of a protein hydrolysate, such as a keratin or collagen hydrolysate. Suitable sugars are glucose, dextrose and fructose. These may be added singly or in the form of, e.g. fruit extracts. A particularly preferred combination of natural hair root nutrients for inclusion in compositions of the invention is isoleucine and glucose. A particularly preferred amino acid nutrient is arginine.

(ii) hair fibre benefit agents. Examples are:

- ceramides, for moisturising the fibre and maintaining cuticle integrity. Ceramides are available by extraction from natural sources, or as synthetic ceramides and pseudoceramides. A preferred ceramide is Ceramide II, ex Quest. Mixtures of ceramides may also be suitable, such as Ceramides LS, ex Laboratoires Serobiologiques.

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- fatty acids, for cuticle repair and damage prevention. Examples are branched chain fatty acids such as 18-methyleicosanoic acid and other homologues of this series, straight chain fatty acids such as stearic, myristic and palmitic acids, and unsaturated fatty acids such as oleic acid, linoleic acid, linolenic acid and arachidonic acid. A preferred fatty acid is oleic acid. The fatty acids may be added singly, as mixtures, or in the form of blends derived from extracts of, e.g. lanolin.

25  
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Mixtures of any of the above active ingredients may also be used. A particularly preferred combination is arginine and oleic acid.

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The invention is further illustrated by way of the following non-limitative Example:

EXAMPLE

5

A hair conditioning composition was prepared by mixing the following components in the amounts stated:

Ingredient

10

		<u>% wt</u>		
	Arquad 16-50	1.4	1.4	1.4
	Laurex CS	1.8	5.0	3.1
15	Paraffin wax	--	--	1.0
	Silicone DC-1766	2.5	--	2.216
	Silicone DC-8177	3.1	10.0	2.063
	Polysurf 67	0.02	--	--
	Preservative	<	qs	>
20	Perfume	0.3	0.3	0.3

Arquad 16-50 is a 50% solution of cetyltrimethylammonium chloride in isopropanol

Laurex CS is a mixture of cetyl and stearyl alcohol

25 Silicone DC-1766 is a 60% emulsion of dimethiconol

Silicone DC-8177 is a 15% emulsion of trimethylsilylamodimethicone with a particle size of 40nm

Polysurf 67 is cetyl hydroxyethylcellulose

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CLAIMS

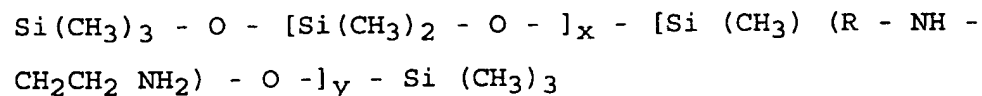
1. An aqueous hair conditioning composition comprising, in addition to water:

5

i) at least one conditioning surfactant, and

ii) emulsified particles of an amino functional silicone of general formula:

10



wherein  $x + y$  is a number from about 50 to about 500, and

15

wherein R is an alkylene group having from 2 to 5 carbon atoms;

in which the amino functional silicone has a mole percent amino functionality of at least 1 mole %.

20

2. A composition according to claim 1, in which the mole percent amino functionality of the amino functional silicone ranges from about 1 to about 8.0 mole %.

25

3. A composition according to claim 1 or claim 2, in which the average amino functional silicone particle size in the hair conditioning composition is less than 2 microns.

30

4. A composition according to any preceding claim, which further comprises emulsified particles of a non-amino functionalised silicone.

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5. A composition according to claim 4, in which the viscosity of the emulsified non-amino functionalised silicone itself is at least 60,000 cst.
- 5 6. A composition according to claim 4 or claim 5, in which the average non-amino functional silicone particle size in the hair conditioning composition is less than 2 microns.
7. A composition according to any preceding claim, which  
10 further comprises a fatty alcohol and/or an alkoxylated fatty alcohol.
8. A composition according to claim 7, in which the weight ratio of cationic surfactant to fatty alcohol and/or  
15 alkoxylated fatty alcohol is from 1:1 to 1:4.
-

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 99/01902

**A. CLASSIFICATION OF SUBJECT MATTER**  
 IPC 6 A61K7/06 A61K7/50

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 152 194 A (PROCTER & GAMBLE) 21 August 1985 (1985-08-21) the whole document ---	1,2,7,8
X	GB 2 157 168 A (L'OREAL ) 23 October 1985 (1985-10-23) page 1-6, line 36 claims 1-20; examples 7,11,15 ---	1,7
X	US 4 586 518 A (CORNWALL SUSAN M ET AL) 6 May 1986 (1986-05-06) cited in the application column 4, line 55-68 column 5, line 1-11 column 6, line 20-22 claims 1,5-9; example 1 --- -/--	1,2

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

13 July 1999

Date of mailing of the international search report

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# INTERNATIONAL SEARCH REPORT

International Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 316 615 A (R & C PRODUCTS PTY LTD) 4 March 1998 (1998-03-04) the whole document ---	1-8
A	M.S. STARCH: "Silicones in Hair Care Products" DRUG AND COSMETIC INDUSTRY, vol. 134, no. 6, 1906 - 1984, XP002109041 pages 38-44 and 102 the whole document -----	1-8

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

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